Residual Gas Analysis in the Spiral Orbit Tribometer

Introduction. The early use of the vacuum SOT to study liquid lubricants used a quadrupole residual gas analyzer (RGA) to observe what were interpreted as fragments of the lubricant's molecules emitted from the rolling /sliding contact. The features observed in the RGA were particularly strong when running fluorocarbon lubricants such as Castrol (Fomblin) or Krytox although not so strong for the hydrocarbon Pennzane 2001A. These features were immediately taken as evidence for the tribochemical destruction (or consumption) of the lubricant with the emission of product fragments into the vacuum ambient where they were detected by the RGA. Other studies used pure sliding pin-on-disk tribometers have also observed features due to tribochemical destruction of the lubricant and they will be commented upon below. The other product of the lubricant's tribochemical destruction was considered to be non-lubricating solid "friction polymer" that remains in or near the track.

The SOT was first used to study the "lifetime" of Fomblin Z-25 on different metals in support of a program for a ball screw on the Chandra X-Ray space telescope. The fluorocarbon fragments observed by the RGA motivated a warning at that time to the Chandra's program managers that such fragments could deposit on adjacent optics and could be considered a threat to the integrity of the telescope. It was recommended that Pennzane (longer lifetime and lower destruction rate) be used instead of Fomblin (shorter lifetime and higher destruction rate). Nevertheless, Fomblin was still chosen and the mechanism it lubricates has worked without failure. However, the Chandra's detector has suffered a loss of sensitivity over the 10 years of operation and the Chandra group's analysis has indicated that the loss is associated with fluorocarbon fragments condensed on the detector. The consensus that has been informally conveyed is that the fluorocarbon fragments have been considered to be liberated from the lubricant by radiation in the orbital environment. Apparently, no considerations were given to tribochemical mechanisms of fragment generation.

Such remains the status to date of RGA use in the SOT. In fact, nothing has ever been published in the open literature on the RGA in the SOT. Although there is a real motivation to use the RGA to definitively identify the emitted fragments as a way to characterize the tribochemical destruction mechanism, there are two limitations on the RGA that must be faced. In the first place, the RGA has a mass range that is much smaller than the molecular weight of the lubricant molecules, so that even if a full molecule was emitted, it couldn't be detected. Second, the RGA employs an electron bombardment ionization source to "crack" the species entering the RGA, thus rendering the interpretation and understanding of the observed features ("cracking pattern") for large molecules very difficult indeed.

Even though the RGA has not been used to answer "fundamental tribochemical" issues, it is so easy to use and the spectral patterns can be so striking that there is good reason to provide the most straightforward of observations from running the SOT with Castrol 815Z (Fomblin Z-25), Krytox 143AC and Pennzane 2001A, simply as a service to SOT users for their possible present and future benefit. In fact, the results will be shown to have some relevance to the systematics of a SOT test even if they are not particularly revealing about tribochemistry itself.

This report will start with a description of the Experimental system followed by the Procedure used in all the tests. The Results will be given for each lubricant in graphical form with simple comments. No real discussion will be given nor will any conclusions be drawn. Excel spreadsheets containing the numeric data that generate the plots shown below are available upon request.

Experimental. SOT III at GRC with an Ametek Dycor Dymaxion RGA was used for the tests. Fig. 1 shows the configuration. A screen on the ionization chamber of the RGA prevents electrons from bombarding and thus damaging the organic liquid lubricants. The RGA has a mass range of 1-100 amu (atomic mass units) and uses a Faraday cup detector. The RGA is controlled by a laptop personal computer (PC) that is independent of the PC that controls and acquires data from the SOT itself. The RGA's PC acquires both analog spectra and time-based intensities (trends) of chosen spectral features. In this report, the features at amu values of 18 (H_2O), 28 (CO), 44 (CO_2), 47 (COF), 66(COF_2), and 69 (CF_3) were acquired. The intensity at 25 amu, a blank part of the spectrum, was also followed for reference purposes.

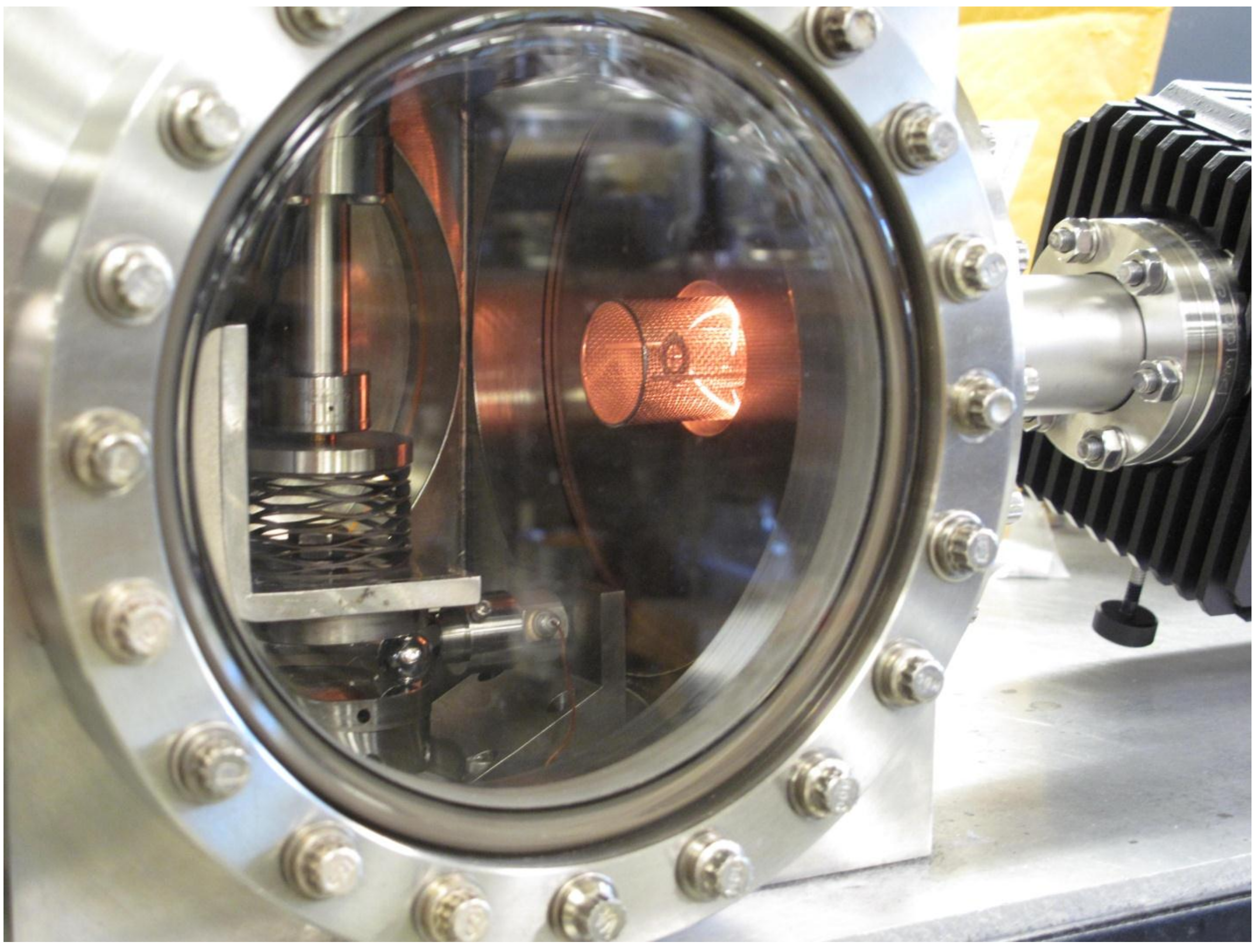

Fig. 1. SOT III with the RGA installed and filament on, and with a ball under load.

All the tests were run at 60 rpm plate speed with 440C steel specimens at a Hertz pressure of 1.5 GPa (43 lb. load on a .5" diameter ball). Lubricant charges on the ball between 20 and 50 μg were used.

Procedure. The system pressure was about $1.5x10^{-8}$ Torr after overnight evacuation. An analog survey spectrum was acquired and then a trend of the chosen features was initiated before rolling started. About midway into the test, trending was stopped and an analog survey spectrum was acquired while still rolling. The spectra displayed below will be *difference* spectra, the difference between the spectrum acquired while rolling minus the spectrum acquired before rolling started. This difference spectrum thus displays only the features generated by rolling. Trending was then resumed until the test concluded or, in the case of Pennzane, the test was manually terminated.

Results for Castrol 815Z. The analog spectrum from a test with Castrol 815Z is shown in Fig.2. The features observed include H_2O, a species not in the lubricant molecule. It turns out that it always appears in such difference spectra no matter what lubricant is used, even with MoS_2. It seems to be a constant artifact whose origin is unknown, although it may be related to the rotary feedthrough operating under load. It does not appear when the feedthrough runs freely under no load. The other features are all related to lubricant's molecular structure, the tribochemically-induced decomposition products or the cracking products of both. As discussed in the Introduction, the cracking pattern is difficult to interpret. In any case, the principle feature is CO_2 with smaller features associated with fluorocarbon species. No feature appears above the noise at 25 amu.

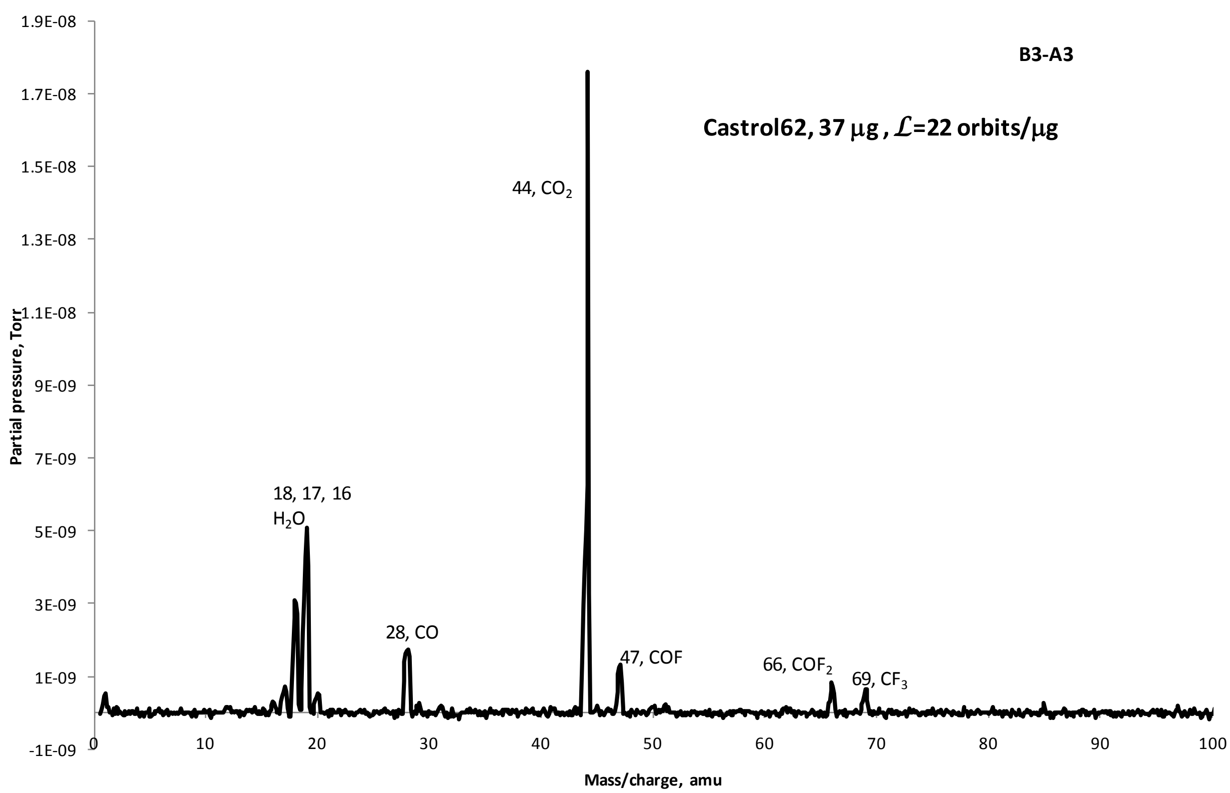

Fig. 2. Analog spectrum for a test with Castrol 815Z

The Trend for the chosen features for running Castrol 815Z as well as the CoF for the friction trace is shown in Fig.3.

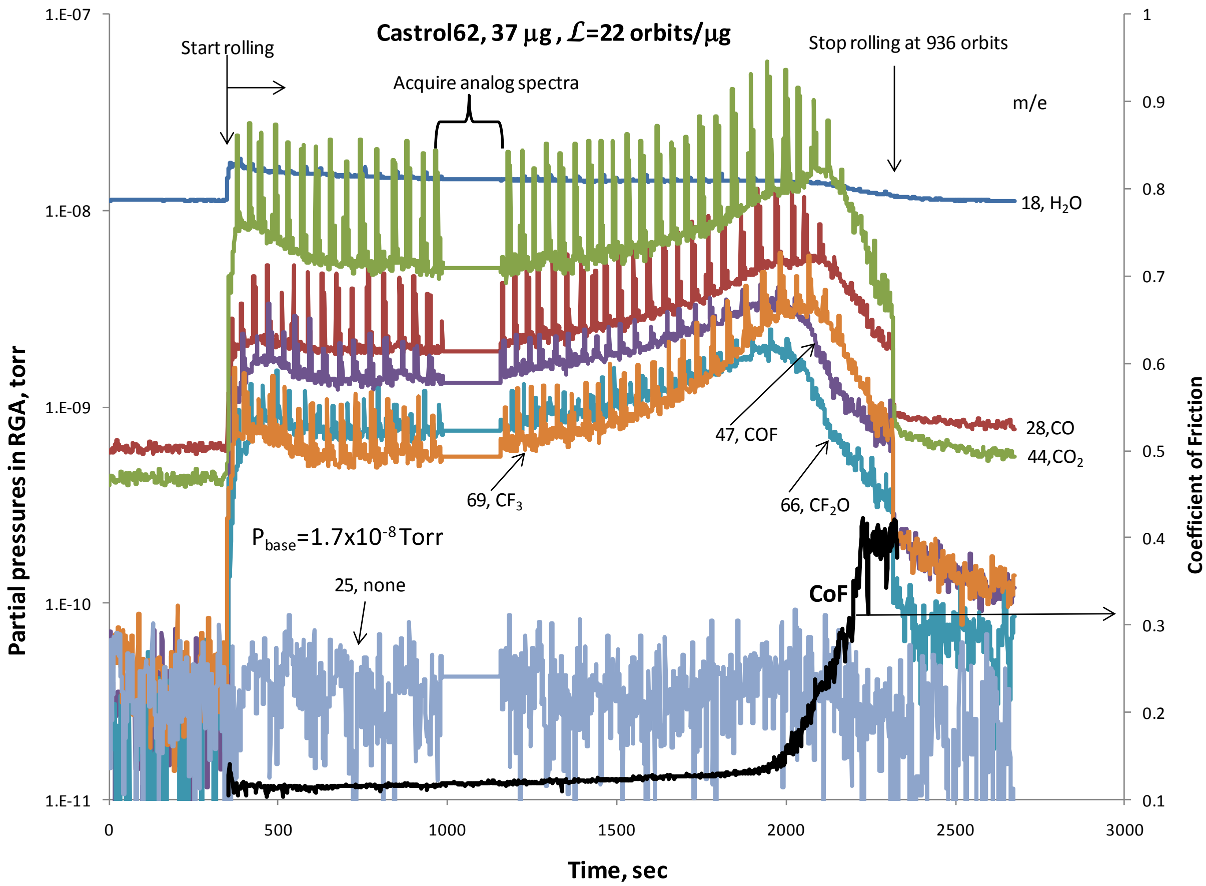

Fig. 3. Trend of intensity of chosen features together with the friction trace for Castrol 815Z

The intensity of all the features, except 25 amu, increase upon the onset of rolling. The sharp spikes in intensity are due to the ball in the scrub and its associated macroscopic sliding, unlike the sliding in the Hertz contact associated with pivoting or spin when the ball rolls outside the scrub. No spikes are observed for the intensity of H_2O, leading to the conclusion that the H_2O is not associated directly with tribochemical destruction of the lubricant molecule. No change in the intensity at 25 amu is observed so that the basic response of the RGA is not changed by virtue of rolling. CO2 is the dominant feature as in the analog spectrum. There is a significant increase in the intensities of the features associated with tribochemical destruction *before* the increase of the CoF near termination of the test because of high CoF. This precursor of failure with liquid lubricants is always observed. When the CoF increases significantly (entering the failure region) the spikes associated with sliding in the scrub disappear. This is consistent with the interpretation that the lubricant has been “used up” and there is no more left to decompose and emit fragments into the ambient to be sensed by the RGA. The general decrease of intensities associated with tribochemical destruction in the friction “failure region” is also consistent with the lack of lubricant – it having been already destroyed. Finally, all intensities (apart from that at 25 amu) decrease sharply at secession of rolling. Some of these aspects show more clearly in Figs. 4a & 4b.

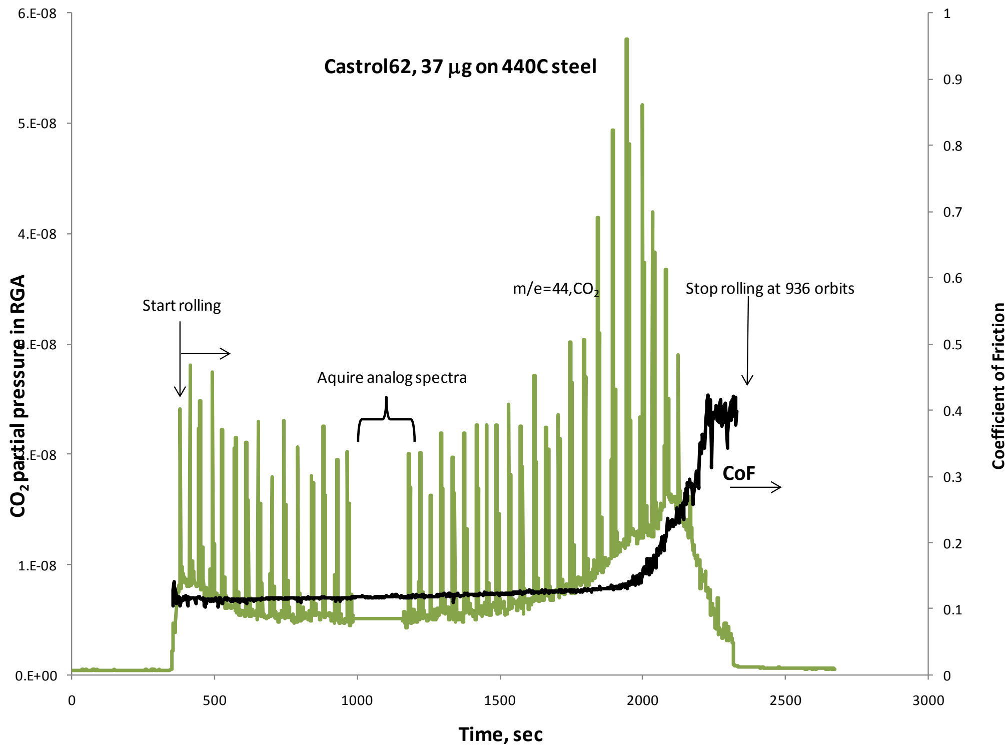

Fig. 4a. Trend of CO_2 intensity and friction trace

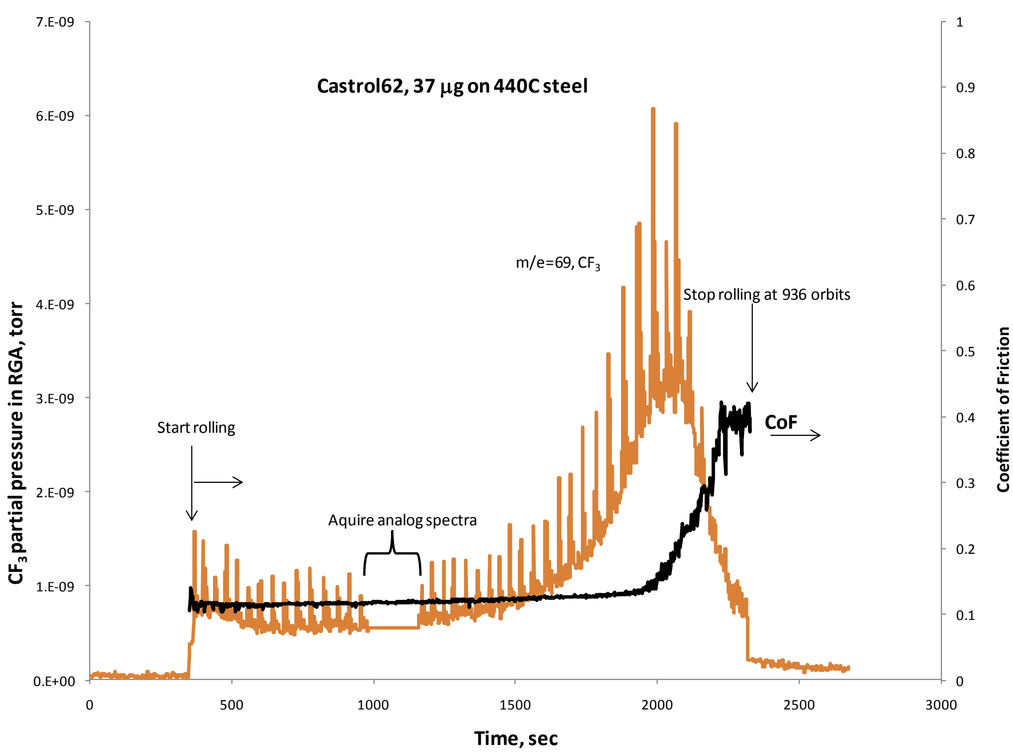

Fig. 4b. Trend of CF_3 intensity and friction trace

Results for Krytox 143AC. The difference spectrum upon rolling on Krytox 143AC is shown in Fig. 5.

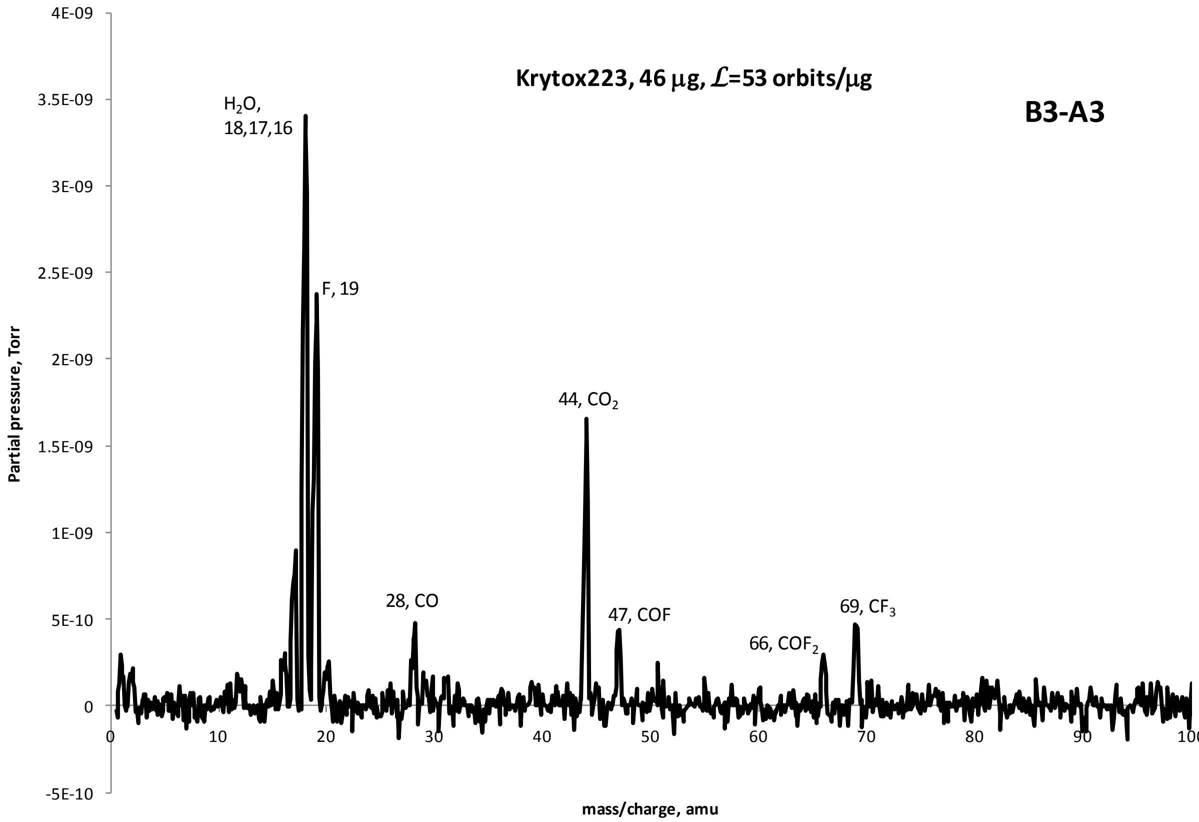

Fig. 5. Analog spectrum for a test with Krytox 143AC

Note first that the overall intensity of this difference spectrum is much lower than the intensity of the spectrum from the test with Castrol 815Z. This is consistent with the longer lifetime of Krytox 143AC relative to that of Castrol 815Z with lower rate of tribochemical destruction and lower rate of emission of fragments. Here the dominant feature is H_2O as opposed to CO_2. The feature at 19 amu is due to fluorine and may be due to contamination of the RGA by previous tests with fluorocarbon lubricants.

The trend of the chosen features and friction trace is shown in Fig. 6. Although CO_2 is again the dominant feature, CF_3 at 69 amu is much stronger than it was in the test for Castrol 815Z, especially near the end of life. This is probably due to the presence of the CF_3 pendant group in the molecular structure of the Krytox molecule that is not present in the Castrol 815Z molecule. There are spikes present in the trends due to sliding in the scrub but they are harder to make out since there is appreciable compression due to the longer lifetime relative to that for Castrol 815Z. Even though CO_2 is dominant, CF_3 becomes very close to domination in the failure region. Another interesting aspect of the behavior is the correlation of the RGA intensities with CoF in the failure region marked with the red asterisk. A decrease of the CoF coincides with a decrease in RGA intensities before the general increase of the CoF and the decrease of the intensities. This is consistent with the idea that it is the friction that forces the tribochemical destruction of the lubricant molecules. The comments made above about the Castrol 815Z tests generally apply to Krytox 143AC.

It should be noted that there is definite evidence here for the emission of tribochemically generated fragments, in contrast to Mori and Morales (see below) who reported no emission of such fragments in sliding experiments with these same two lubricants.

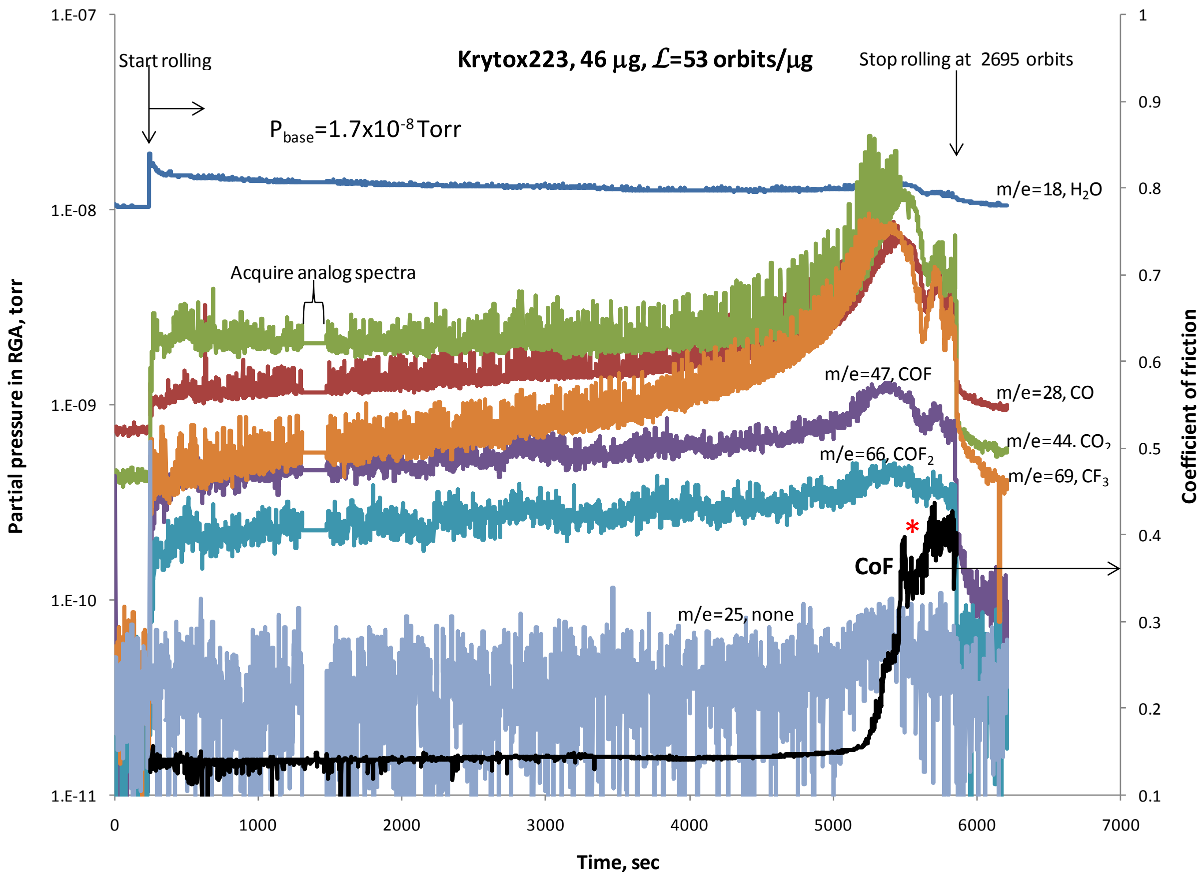

Fig. 6. Trend of intensity of chosen features together with the friction trace for Krytox 143AC

Results for Pennzane 2001A. The analog spectrum for the test rolling on Pennzane 2001A is shown in Fig. 7 below. The spectrum is dominated by H_2O, as was the spectrum for Krytox 143AC. Again, this must be due to the low rate of fragment emission associated with the very long life of Pennzane 2001A relative to that of Castrol 815Z. Even though the Pennzane 2001A molecule contains no fluorine, there are still fluorine features at 19 amu and 47 amu that are attributed here to contamination as mentioned above.

The trends of the chosen features together with the friction trace are shown in Fig. 8. Note first that the test was manually terminated after 900 orbits. There are increases for the fluorine-containing features at 47 amu, 66 amu and 69 amu that, again, are attributed to contamination from previous tests with lubricants containing fluorine. These features have low intensity and appreciable noise, so it is not easy

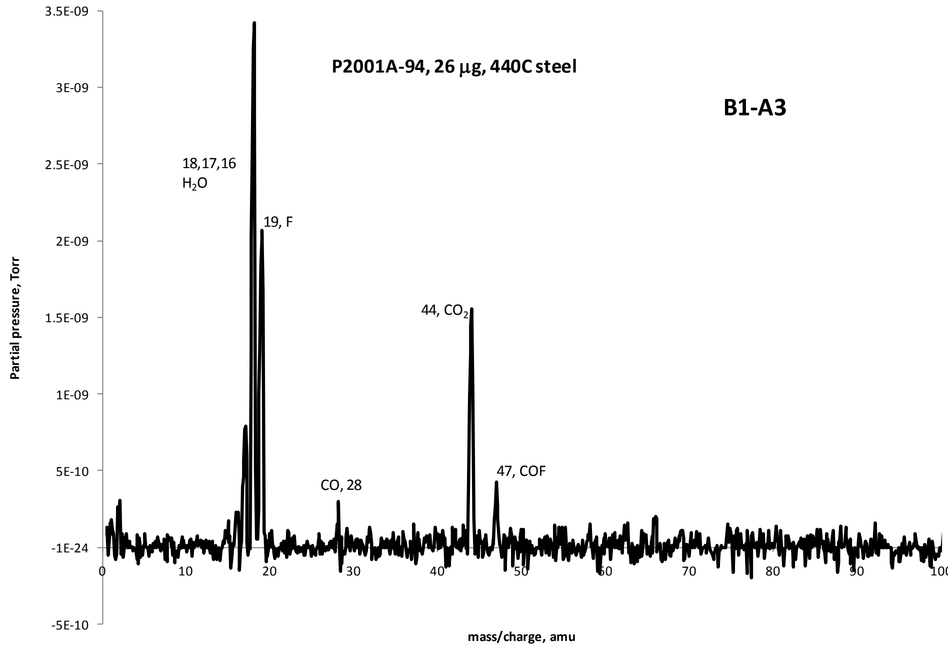

Fig. 7. Analog spectrum for a test with Pennzane 2001A

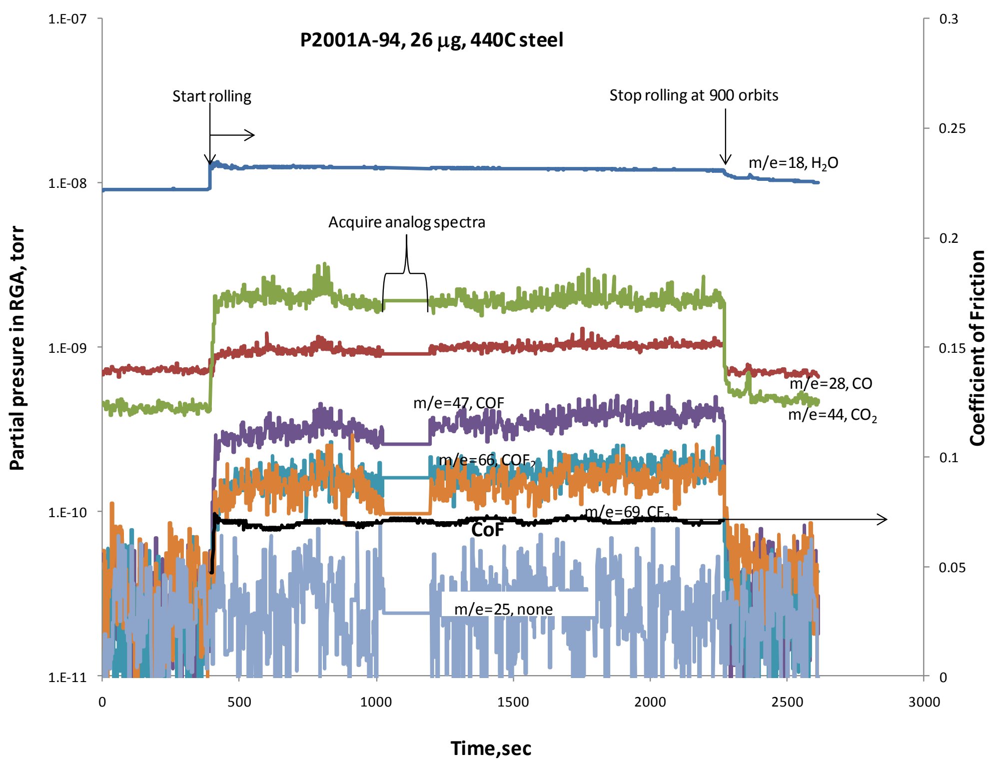

Fig. 8. Trend of intensity of chosen features together with the friction trace for Pennzane 2001A

to determine if there are scrub-related spikes present in these trends. They're probably not present and really shouldn't be present for this lubricant. However, true spikes appear to be present for CO_2, but not for CO. So these results for Pennzane 2001A are dominated by artifacts – the fluorocarbons, H_2O and CO – which are not really representative of the tribochemical destruction of the Pennzane molecule.

Caution. An RGA to study tribochemical destruction of lubricant molecules should be used with caution since the artifacts seen here may often appear when least expected. In particular, they have appeared when running MoS_2 in the SOT. But to return to the issue about tribochemically-generated fragments depositing on sensitive surfaces on spacecraft – it is pretty clear that the use of a simple RGA such as the one used here is quite limited in addressing that issue. Perhaps an RGA with greater mass range or higher sensitivity will be more useful. But at the very least an RGA can indicate that *something* is escaping the tribological contact and that alone may be cause for concern in some applications. Perhaps future studies with a more capable RGA will shed more light on tribochemical reaction pathways.

Some Papers :

- Mori and Morales, Trib. Trans., Vol. 33, p. 325, 1990
- Strom et al., Wear, Vol. 168, p. 31, 1993
- Lu et al., Trib. Letters, Vol. 27, p. 25, 2007
- Mori and Morales, Wear, Vol. 132, p. 111, 1989
- John et al., Trib. Letters, Vol. 9, p. 167, 2000